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APPLICATION NUMBER: *60/553,659*
FILING DATE: *March 16, 2004*
RELATED PCT APPLICATION NUMBER: *PCT/US05/08458*

Certified by

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PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

Express Mail Label No. ET122668029US

INVENTOR(S)

Given Name (first and middle [if any])	Family Name or Surname	Residence (City and either State or Foreign Country)
Dany Norwin	De Kock Van Riel	Assenede, Belgium Aardenburg, The Netherlands

☐ *Additional inventors are being named on th____ separately numbered sheets attached hereto*

TITLE OF THE INVENTION (500 characters max)

METHOD FOR PREPARING LONG GLASS FIBER-REINFORCED COMPOSITION AND FABRICATED ARTICLES THEREFROM

Direct all correspondence to: **CORRESPONDENCE ADDRESS**

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ENCLOSED APPLICATION PARTS *(check all that apply)*

☒ Specification *Number of Pages* 16 ☐ CD(s), Number

☐ Drawing(s) *Number of Sheets* ☒ Other (specify) Express Mailing Certificate, Fee Transmittal for FY 2004, postcard

☐ Application Data Sheet. See 37 CFR 1.76

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☐ Applicant claims small entity status. See 37 CFR 1.27.

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Respectfully submitted,

SIGNATURE MA Schaldenbrand for TTM

Date 03/16/04

TYPED or PRINTED NAME Michael A. Schaldenbrand

REGISTRATION NO. 47,923 *(if appropriate)*

Docket Number: 25289-0025

TELEPHONE (248) 433-7570

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FEE TRANSMITTAL for FY 2004

Effective 10/01/2003. Patent fees are subject to annual revision.

☐ Applicant claims small entity status. See 37 CFR 1.27

TOTAL AMOUNT OF PAYMENT x > $ **$160.00**

Complete if Known	
Application Number	
Filing Date	
First Named Inventor	Dany De Kock
Examiner Name	
Art Unit	
Attorney Docket No.	25289-0025

METHOD OF PAYMENT *(check all that apply)*

☒ Check ☐ Credit card ☐ Money Order ☐ Other ☐ None

☒ Deposit Account:

Deposit Account Number: 04-1061

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FEE CALCULATION

1. BASIC FILING FEE

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description	Fee Paid
1001	770	2001	385	Utility filing fee	
1002	340	2002	170	Design filing fee	
1003	530	2003	265	Plant filing fee	
1004	770	2004	385	Reissue filing fee	
1005	160	2005	80	Provisional filing fee	160.00

SUBTOTAL (1) x > $ **$160.00**

2. EXTRA CLAIM FEES FOR UTILITY AND

	Extra Claims		Fee from below		Fee Paid
Total Claims		-20** = 0	X	=	0.00
Independent Claims		- 3** = 0	X	=	0.00
Multiple Dependent				=	

Large Entity Fee Code	Large Entity Fee ($)	Small Entity Fee Code	Small Entity Fee ($)	Fee Description
1202	18	2202	9	Claims in excess of 20
1201	86	2201	43	Independent claims in excess of 3
1203	290	2203	145	Multiple dependent claim, if not paid
1204	86	2204	43	** Reissue independent claims over original patent
1205	18	2205	9	** Reissue claims in excess of 20 and over original patent

SUBTOTAL (2) x > $ **$0.00**

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FEE CALCULATION (continued)

3. ADDITIONAL FEES

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1051	130	2051	65	Surcharge - late filing fee or oath	
1052	50	2052	25	Surcharge - late provisional filing fee or cover sheet	
1053	130	1053	130	Non - English specification	
1812	2,520	1812	2,520	For filing a request for *ex parte* reexamination	
1804	920*	1804	920*	Requesting publication of SIR prior to Examiner action	
1805	1,840*	1805	1,840*	Requesting publication of SIR after Examiner action	
1251	110	2251	55	Extension for reply within first month	
1252	420	2252	210	Extension for reply within second month	
1253	950	2253	475	Extension for reply within third month	
1254	1,480	2254	740	Extension for reply within fourth month	
1255	2,010	2255	1,005	Extension for reply within fifth month	
1401	330	2401	165	Notice of Appeal	
1402	330	2402	165	Filing a brief in support of an appeal	
1403	290	2403	145	Request for oral hearing	
1451	1,510	1451	1,510	Petition to institute a public use proceeding	
1452	110	2452	55	Petition to revive - unavoidable	
1453	1,330	2453	665	Petition to revive - unintentional	
1501	1,330	2501	665	Utility issue fee (or reissue)	
1502	480	2502	240	Design issue fee	
1503	640	2503	320	Plant issue fee	
1460	130	1460	130	Petitions to the Commissioner	
1807	50	1807	50	Processing fee under 37 CFR § 1.17(q)	
1806	180	1806	180	Submission of Information Disclosure Statement	
8021	40	8021	40	Recording each patent assignment per property (times number of properties)	
1809	770	2809	385	Filing a submission after final rejection (37 CFR § 1.129(a))	
1810	770	2810	385	For each additional invention to be examined (37 CFR § 1.129(b))	
1801	770	2801	385	Request for Continued Examination (RCE)	
1802	900	1802	900	Request for expedited examination of a design application	

Other fee (specify) _____

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SUBTOTAL (3) x > $

SUBMITTED BY

				Complete *(if applicable)*	
Name (Print/Type)	Michael A. Schaldenbrand	*Registration No. (Attorney/Agent)*	47,923	*Telephone*	(248) 433-7570
Signature	Michael A. Schaldenbrand for TTM			*Date*	March 16, 2004

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CERTIFICATE OF MAILING BY "EXPRESS MAIL" (37 CFR 1.10) Applicant(s): **Dany De Kock and Norwin Van Riel**	Docket No. **25289-0025**

Serial No.	Filing Date	Examiner	Group Art Unit

Invention: **METHOD FOR PREPARING LONG GLASS FIBER-REINFORCED COMPOSITION AND FABRICATED ARTICLES THEREFROM**

I hereby certify that the following correspondence:

PROVISIONAL PATENT APPLICATION including return receipt postcard, Check No. 34549 for $160.00, Provisional Application for Patent Cover Sheet (1 page), Fee Transmittal for FY 2004 (1 page, in duplicate) and 16 page provisional patent application

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METHOD FOR PREPARING LONG GLASS FIBER-REINFORCED COMPOSITION AND FABRICATED ARTICLES THEREFROM

FIELD OF THE INVENTION

The present invention concerns a process for preparing a long fiber glass-filled thermoplastic composition and fabricated articles therefrom.

BACKGROUND OF THE INVENTION

It is well known that the physical properties of thermoplastics can be improved by the incorporation of filler materials such as glass fibers. The incorporation of reinforcing fibers into polymeric products beneficially affects resin properties such as tensile strength, stiffness, dimensional stability and resistance to creep and thermal expansion. Traditional methods of producing such articles have been through use in standard, pre-compounded short fiber glass-filled ABS. While satisfying certain objectives in optimizing the quality of the finished product, conventional methods have proven to be commercially costly and in other ways have fallen short of their objectives in terms of density, impact performance and strength. A lower cost solution to the known methods of producing fiber-reinforced articles is desired.

Certain steps have been taken in overcoming the deficiencies of known methods by incorporating long glass fibers into thermoplastic material for producing a long fiber-reinforced thermoplastic article. In this regard reference may be had to WO 01/02471, titled LONG FIBER-REINFORCED THERMOPLASTIC MATERIAL AND METHOD FOR PRODUCING THE SAME. According to this reference, long glass fibers are impregnated with a first thermoplastic material. The matrix of the material is composed of at least two different thermoplastics, thus enabling the fibers to be wet by one of the two thermoplastic materials. The resulting article demonstrates improved physical, chemical and electrochemical properties. However, while demonstrating an improvement in the state of technology, the process set forth in WO 01/02471 is

burdened by the requirement to employ at least two thermoplastics for production of the glass fiber reinforced granulate.

Reference may also be had to WO 0003852, titled GRANULES FOR THE PRODUCTION OF A MOLDING WITH A CLASS-A SURFACE, PROCESS FOR THE PRODUCTION OF GRANULES AND ITS USE. According to this reference, a granulate for the production of Class-A surface moldings is provided. The granulate comprises a thermoplastic polymer and long fiber material. The fiber material is provided with lengths in the range of 1 to 25 mm. While also demonstrating an improvement in the state of technology, this reference is limited in its application to articles requiring Class-A surfaces and, furthermore, is limited by its inherent inability to achieve performance benefits realized through the use of amorphous polymers.

Further reference may be made to U.S. Patent No. 5,783,129, titled APPARATUS, METHOD, AND COATING DIE FOR PRODUCING LONG FIBER-REINFORCED THERMOPLASTIC RESIN COMPOSITION. According to this reference a method is disclosed for producing a long fiber-reinforced thermoplastic resin composition composed of a thermoplastic resin and fiber bundles. The preferred resins are selected from the group which includes semi-crystalline polymers like polyolefins, polyesters, and polyamides. A reference to the same assignee, U.S. Patent No. 5,788,908 for METHOD FOR PRODUCING FIBER-REINFORCED THERMOPLASTIC RESIN COMPOSITION, is similar in that it too discloses a method for producing long fiber-reinforced thermoplastic resin composition. According to the disclosed method of production, a web-like continuous fiber bundle is impregnated with a thermoplastic resin melt to form a composite material. As with the preceding reference, the preferred resins

are selected from the group which includes semi-crystalline polymers like polyolefins, polyesters, and polyamides. While these methods provide certain advantages over the prior art, the products produced by these methods are not able to demonstrate desired dimensional performance.

It would therefore be desirable to find an efficient and effective means of producing long glass fiber-reinforced articles that demonstrate lowered density, improved impact properties, improved strength properties, and superior dimensional stability as achieved with amorphous polymers but at reduced production costs.

SUMMARY OF THE INVENTION

The present invention addresses the deficiencies of the art by providing a process for preparing a superior long glass fiber-reinforced composition for the production of a glass fiber-reinforced article of manufacture generally comprising:

(a) Selecting a quantity of long glass fiber;

(b) adding the selected quantity of long glass fiber to a first copolymer to form a master-batch, the first copolymer being a high flow copolymer; and

(c) blending the master-batch with a second copolymer, the second copolymer being a stiffer flowing amorphous styrenic copolymer.

The first copolymer, the high flow copolymer, is preferably styrene-acrylonitrile (SAN), although others polymers may be used in addition to or in lieu thereof when forming a homogeneous blend with the stiffer flowing amorphous styrenic copolymer. The second copolymer, the stiffer flowing styrenic copolymer, is acrylonitrile-butadiene-styrene (ABS), although others may be used in addition to or in lieu thereof. The master-batch is preferably dry-blended or is dosed by the use of a mixing unit with the second styrenic copolymer.

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DETAILED DESCRIPTION OF THE INVENTION

The present invention provides a process for the preparation of a superior long fiber glass-filled thermoplastic composition for use in the production of a molded article that demonstrates high dimensional stability. The method for producing the composition of the present invention offers a low-cost approach to the production of a moldable compound having low density and high impact strength when compared to products produced by known methods.

The process of the present invention for the preparation of a fiber-reinforced product comprises the general steps of selecting a quantity of long glass fiber, adding the selected quantity of long glass fiber to a high flow of a first copolymer to form a master-batch, blending the master-batch with a second stiffer flowing styrenic copolymer to form an injection moldable or compression moldable glass fiber-reinforced resin compound, injecting the resin compound into a mold, and recovering a fiber-reinforced polymerized part.

The targeted fiber length in the master-batch is between 3.0 mm and 25.0 mm with an average length of about 15.0 mm. Long glass fibers or a plurality of glass strands bundled in the form of widely-used glass roving may be incorporated. Specific glass rovings may be used for particular applications. In any event, typically the glass fibers will be substantially uniform in length, with the length dependent upon the granule size of the long glass fiber master-batch.

The targeted average fiber length in the final product is between 0.5 mm and 25.0 mm with a preferred average length between 3.0 mm and 8.0 mm when injected molded. In the event that the pellets are compression molded, the fiber length will be

related to the size of the pellets, *e.g.*, the fiber length would be between 11.0 mm and 25.0 mm.

The glass fibers are added to a flow of a carrier melt. The carrier is a high flow copolymer which provides sufficient wetting and reduced shear forces on the glass fibers to avoid uncontrolled sizing but sufficient dispersion. The carrier material is a high flow version of, or forms a homogenous mixture with, the second stiffer flowing unreinforced amorphous unfilled material. The carrier may consist of both amorphous or functionalized semi-crystalline materials or blends thereof. Preferably the carrier is a styrene-acrylonitrile (SAN) such as Tyril ® (trademark, Dow Chemical Company) or acrylonitrile-butadiene-styrene (ABS) such as MAGNUM ® (trademark, Dow Chemical Company) or a styrene-maleic anhydride (SMA) such as DYLARC ® (trademark, Arco Chemical Company). As a variation to the use of a styrenic-based carrier, alternate high flow versions engineering thermoplastic resins may be used or blended with the styrenic-based carrier such as polycarbonate (PC) such as CALIBRE ® (trademark, Dow Chemical Company) or a thermoplastic polyurethane such as ISOPLAST ® (trademark, Dow Chemical Company).

Although there are alternative methods for adding the glass fibers to the carrier flow, the glass fiber may be added to the high flow carrier melt by way of a side feeder of the compounding unit. Preferably the glass fiber is added to the high flow carrier melt in such an amount so that sufficient wetting and dispersion is achievable. A glass fiber concentration of 75% is possible but may provide a high vulnerability to poor dispersion. Preferably the glass fiber concentration is in the range of between about 40% and 60%

with the overall objective being to provide as high a concentration as possible while minimizing poor dispersion.

Once the master-batch is formed it is dry-blended with the stiffer flowing unreinforced, second amorphous copolymer. Preferably the second unreinforced amorphous material is a styrenic copolymer such as an acrylate styrene acrylonitrile (ASA), ABS, or SMA. This neat polymer will contribute to the strength and heat of the final blend. By use of the master-batch concept, the high level performance of the second polymer is not compromised with additional material characteristics as required for a high dosing level LG fiber reinforcing process.

The addition level of the master-batch is between about 10% and about 40% depending on the required stiffness and dimensional performance of the final article.

The resulting dry blend is injected molded under standard injection conditions for the second non-reinforced polymer into a mold. The resulting glass fiber-reinforced article is thereafter removed from the mold.

A broad variety of additives may be included in the thermoplastic resins set forth above according to the specific applications and use of the resin composition. Such additives may include one or more of colorants, de-molding agents, anti-oxidants, UV stabilizers or inorganic fillers

In general, a fiber-reinforced molded article produced according to the method fo the present invention achieved several unexpected results. Of these results it was found that fewer glass fibers were needed to obtain a similar heat performance when compared with articles prepared according to known methods. It was also found that the resulting article had lower density and reduced weight when compared with such

articles. Furthermore, the resulting article demonstrated improved impact performance, strength levels and heat resistance (at equivalent levels of stiffness) over articles produced according to known methods.

The process of the present invention is illustrated by the following practical example and comparative testing wherein all parts and percentages are by volume unless otherwise specified.

PRACTICAL EXAMPLE

A long glass fiber master-batch is prepared using glass roving added, via a pultrusion or co-extrusion process, into a high flow SAN melt. The obtained glass fiber content in the master-batch was between 55% and 60%. This master-batch was dry-blended with several neat mass ABS resins in blending ratios between 15% and 35%. The dry-blend was used for molding articles in an injection molding machine under standard ABS conditions into an ISO test specimen.

COMPARATIVE TESTING

The table below shows the obtained physical properties for three different dry blends prepared in accordance with the practical example set forth above with the exception of specified variations in glass levels in the master-batch and targeted glass fiber levels. Comparisons were made with a commercially available 16% short glass fiber containing ABS (Reference 1) compound and a commercially available 17% short glass fiber containing ABS (Reference 2).

	Load	neat ABS grade	Sample 1 MAGNUM® 3404	Sample 2 MAGNUM® 3404	Sample 3 MAGNUM® 3416	Reference 1	Reference 2
Norm	Unit	Addition lvl LFG MB	26%	35%	30%	0	0
		Targeted Glass lvl	15%	20%	17%	16%	17%
	kg/l	Density	1.145	1.191	1.16	1.16	1.17
	%	ash content	13.8	19	16	16	
ISO 178	MPa	Flex.mod.(regr.0.05-0.25%)	5279	5910	6201	5519	4700
ISO 178	MPa	Flex strength	134	145	150	103	90
ISO 527-2	MPa	Tensile yield	88	99	99	74	65
ISO 527-2	%	Elongation at rupture	2.3	1.9	2.1	1.7	
ISO 527-2	MPa	Regr. modulus (0.05-0.25%)	4810	6200	5857	5575	5100
ISO 179/1f	kJ/m^2	Unnotched Charpy impact 23°C	23.2	22.8	24.5	18	
ISO 179/1e	kJ/m^2	Notched Izod impact 23°C	14.2	14.6	14.2	6	7
ISO 75A	°C	HDT 1.8MPa	104	119	109	102	96
ISO 306	°C	Vicat 50°C/hr 5kg	106	110	113	106	101
ISO6603-2	J	Total energy	8.5	8.8	8.2	4.6	

"Magnum" is a registered trademark of the Dow Chemical Company.

As the comparative results illustrate, the articles produced according to the composition and method of the present invention demonstrate superior qualities in several areas, including reduced density, increased modulus, increased strength, improved notched impact strength and practical toughness and improved heat resistance.

It is understood that the above are merely preferred embodiments and that various changes and alterations can be made without departing from the spirit and broader aspects of the invention.

CLAIMS

What is claimed is:

1. A method for producing a long glass fiber-reinforced thermoplastic resin composition, the method comprising the steps of:

selecting a quantity of long glass fiber;

adding the selected quantity of long glass fiber to a first copolymer to form a master-batch, said first copolymer being a high flow copolymer; and

blending the master-batch with a second copolymer.

2. The method in accordance with Claim 1 wherein the first and second copolymers are styrenic copolymers.

3. The method in accordance with Claim 1 wherein the first copolymer is selected from the group consisting of styrene-acrylonitrile (SAN), acrylonitrile-butadiene-styrene (ABS), and an alloy of ABS resins.

4. The method in accordance with Claim 1 wherein the second copolymer is a stiffer flowing material selected from the group consisting of acrylonitrile-butadiene-styrene (ABS), styrene-maleic anhydride (SMA), acrylate styrene acrylonitrile (ASA), and alloys thereof.

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5. The method in accordance with Claim 1 wherein the second copolymer is a stiffer flowing material and blends with the first copolymer to form a homogeneous blend.

6. The method in accordance with Claim 1 wherein the second copolymer is a stiffer flowing amorphous styrenic copolymer.

7. The method in accordance with Claim 1 wherein the selected quantity of glass fibers is added to a high flow of the first copolymer.

8. The method in accordance with Claim 1 wherein the selected quantity of glass fibers is added to the first copolymer in such an amount so that the resulting master-batch has a glass fiber concentration of between about 40% and 75%.

9. The method in accordance with Claim 1 wherein the blending ratio of the master-batch with the second copolymer is between about 10% and about 40%.

10. The method in accordance with Claim 1 wherein the long glass fiber is glass roving.

11. The method in accordance with Claim 1 wherein the master-batch is dry-blended with the second copolymer.

12. The method in accordance with Claim 1 wherein the second copolymer is a neat mass acrylonitrile-butadiene-styrene (ABS) resin.

13. A method for producing a long glass fiber-reinforced thermoplastic resin composition, the method comprising the steps of:

selecting a quantity of long glass fiber;

adding the selected quantity of long glass fiber to a first copolymer to form a master-batch, the first copolymer being selected from the group consisting of styrene-acrylonitrile (SAN), acrylonitrile-butadiene-styrene (ABS), an alloy of ABS resins, and polycarbonate; and

dry blending the master-batch with a second copolymer selected from the group consisting of acrylonitrile-butadiene-styrene (ABS), styrene-maleic anhydride (SMA), acrylate styrene acrylonitrile (ASA), and alloys thereof.

14. The method in accordance with Claim 13 wherein the first copolymer is a high flow copolymer.

15. The method in accordance with Claim 13 wherein the second copolymer is a stiffer flowing material and blends with the first copolymer to form a homogeneous blend.

16. The method in accordance with Claim 13 wherein the selected quantity of glass fibers is added to a high flow of the first copolymer.

17. The method in accordance with Claim 13 wherein the selected quantity of glass fibers is added to the first copolymer in such an amount so that the resulting master-batch has a glass fiber concentration of between about 40% and about 75%.

18. The method in accordance with Claim 13 wherein the blending ratio of the master-batch with the second copolymer is between about 10% and about 40%.

19. The method in accordance with Claim 13 wherein the long glass fiber is glass roving.

20. A glass fiber-reinforced article manufactured by the process comprising:

adding a quantity of long glass fiber to a first copolymer to form a master-batch, the first copolymer being a high flow copolymer selected from the group consisting of styrene-acrylonitrile (SAN), acrylonitrile-butadiene-styrene (ABS), an alloy of ABS resins, and polycarbonate;

blending the master-batch with a second copolymer selected from the group consisting of acrylonitrile-butadiene-styrene (ABS), styrene-maleic anhydride (SMA), acrylate styrene acrylonitrile (ASA), and alloys thereof to form an injectable composition; and

injecting the composition into a mold.

ABSTRACT

Process for production of a long fiber glass-filled ABS comprising (a) forming a long glass fiber master-batch by adding a long glass fiber to a high flow styrene-acrylonitrile (SAN) copolymer and (b) blending the master-batch with neat mass ABS resin. A molded article demonstrating high dimensional stability, good impact, strength and heat performance is obtained.

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